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IS 12921 (1990): Lac dye [CHD 23: Lac, Lac Products and Polishes]

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IS 12921 : 1990

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Indian Standard

LAC DYE — SPECIFICATION

भारतीय मानक

लाख रंजक — विशिष्टि

UDC 667·271·3

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 1990

Price Group 2

Lac and Lac Products Sectional Committee, CHD 22

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 15 February 1990, after the draft finalized by the Lac and Lac Products Sectional Committee had been approved by the Chemical Division Council.

Lac dye is available in the stick lac or crude lac but is essentially an integral part of the lac insect *Kerria Lacca* which grows on certain plants. Presently, it is used in some countries like Japan and China in the preparation of soft drinks, bean jam and ham sausage. Its demand is likely to grow substantially in future particularly in food industry including beverages, sweets, confectionery, drugs and cosmetics.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

LAC DYE — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for lac dye.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
323 : 1959	Rectified spirit
1699 : 1974	Methods of sampling and test for food colours (*first revision*)
4905 : 1968	Methods for random sampling
4908 : 1968	Glossary of terms used in lac industry
5741 : 1970	Methods for determination of *p*H
5762 : 1970	Methods for determination of melting point and melting range
6921 : 1973	Methods of sampling and test for lac and lac products

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given in IS 4908 : 1968 shall apply.

4 REQUIREMENTS

4.1 The material shall be in the form of powder or crystals.

4.2 The product shall also comply with the requirements given in Table 1 when tested by the methods prescribed in col 4 of Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be suitably packed, in plastic or glass containers so as to avoid its contact with air.

5.2 Marking

The containers shall be securely closed and bear legibly and indelibly the following particulars:

a) Name of the material,
b) Indication of the source of manufacture;
c) Gross and net mass,
d) Year and date of manufacture, and
e) Batch number or lot number in code or otherwise.

Table 1 Requirements for Lac Dye

(*Clause* 4.2)

Sl No.	Characteristic	Requirements	Method of Test, Ref to	
			Clause No. in Annex A	Clause No. and IS No.
(1)	(2)	(3)	(4)	(5)
i)	Melting point, °C, *Max*	230 (with charring)	A-1	—
ii)	Volatile matter at 135°C, percent by mass, *Max*	0·1	A-2	—
iii)	Ash content, percent by mass, *Max*	0·7	A-3	—
iv)	Cold water insoluble matter, percent by mass, *Max*	52	A-4	—
v)	Hot water insoluble matter, percent by mass, *Max*	10	A-5	—
vi)	Hot alcohol insoluble matter, percent by mass, *Max*	1	—	6 of IS 6921: 1973
vii)	Acid value	234-245	A-6	—
viii)	Dye content, percent by mass, *Min*	90	A-7	—
ix)	UV - visible absorption spectrum positions of absorption, *Max*	230, 292, 340, 500 and 530 nm	A-8	—
x)	Lead, mg/kg, *Max*	10	—	9 of IS 1699: 1974
xi)	Arsenic, mg/kg, *Max*	3	—	10 of IS 1699: 1974

6 SAMPLING

6.1 The method of drawing representative samples of the product and the criteria for conformity shall be as prescribed in Annex B.

ANNEX A

(*Clause* 4.2 and *Table* 1)

METHODS OF TESTS FOR LAC DYE

A-1 DETERMINATION OF MELTING POINT

A-1.1 The melting point of the sample shall be determined by the method given in IS 5762 : 1970.

A-2 DETERMINATION OF VOLATILE MATTER AT 135°C

A-2.1 Apparatus

a) Oven

b) Petri dish (Pyrex or equivalent), 75 mm diameter.

A-2.2 Procedure

Weigh accurately about 1 g of the sample in a petri dish previously dried and weighed, and heat for 3 hours in an air oven maintained at 135 ± 2°C. Cool the petri dish in a desiccator and weigh. Repeat the process until the difference between two successive weighings is less than one milligram. Record the lowest mass obtained.

A-2.3 Calculation

$$\text{Volatile matter, percent by mass} = \frac{100\ (M_1 - M_2)}{M_1 - M}$$

where

M_1 = mass in g of the petri dish with the material before heating,

M_2 = mass in g of the petri dish with material after heating, and

M = mass in g of the empty petri dish.

A-3 DETERMINATION OF ASH CONTENT

A-3.1 Apparatus

a) *Muffle Furnace*

b) *Crucible*, 50-ml capacity.

A-3.2 Procedure

Weigh accurately 1 g of lac dye in a previously weighed crucible and place it in a muffle furnace maintained at 300°C for one hour. Raise the temperature of the furnace to 600°C and keep it at 600°C for one hour. Cool the crucible to room temperature in a desiccator and weigh. Repeat heating in muffle furnace at 600°C for one hour, cooling and weighing until the difference between two successive weighings is less than one milligram.

A-3.3 Calculation

$$\text{Ash, percent by mass} = \frac{100\ M_2}{M_1}$$

where

M_2 = mass in g of ash, and

M_1 = mass in g of sample taken for the test.

A-4 DETERMINATION OF COLD WATER INSOLUBLE MATTER

A-4.1 Procedure

Take accurately about 1 g of lac dye in a beaker. Add 200 ml of water and stir thoroughly. Cover the beaker with a watch glass and allow to stand for 4 hours at 27 ± 2°C with occasional stirring. Filter into a filter flask through a tared and dried sintered glass crucible (G-3). Wash it several times with water. Dry the crucible with the residue at 100 ± 2°C to a constant mass.

A-4.2 Calculation

$$\text{Matter insoluble in cold water, percent by mass} = \frac{m}{M} \times 100$$

where

m = mass in g of residue, and

M = mass in g of the sample taken for test.

A-5 DETERMINATION OF HOT WATER INSOLUBLE WATER

A-5.1 Procedure

Take accurately about 1 g of the material in a beaker and add 200 ml of water. Heat to a temperature of 60°C on a water bath with stirring for half an hour. Filter hot through a tared and dried sintered glass crucible (G-3). Wash several times with hot water. Dry the crucible with the residue at 100 ± 2°C to a constant mass.

A-5.2 Calculation

$$\text{Matter insoluble in hot water, percent by mass} = \frac{m}{M} \times 100$$

where

m = mass in g of residue, and

M = mass in g of the sample taken for test.

A-6 DETERMINATION OF ACID VALUE

A-6.1 Following two methods are recommended:

a) Conventional titration with standard alkali, and

b) Titration using a *p*H meter.

A-6.2 Apparatus

a) Burettes, pipettes, volumetric flask, beakers.

b) *p*H meter — (*See* IS 5741 : 1970).

A-6.2.1 *Reagents*

a) Sodium hydroxide solution, 0·1 *N*;

b) Phenolphthalein indicator; and

c) Thymol blue indicator.

A-6.3 Procedure

A-6.3.1 *Preparation of 0·1 Percent Aqueous Dye Solution*

Weigh accurately 0·5 g of powdered lac dye in a 100-ml beaker. Add 50 ml of distilled water and heat on a water bath up to 90°C for 10 minutes. Decant the clear solution into a 500-ml measuring flask. Again add 50 ml of distilled water and heat on a water bath and decant the clear solution as before in the measuring flask. Repeat this till the dye goes into the solution. Allow the solution in the measuring flask to cool to room temperature and make the volume up to the mark. Filter under vacuum, if necessary, through sintered Gooch crucible funnel into a thoroughly washed and dried filtering flask. Transfer the clear dye solution into a washed and dried reagent bottle.

This is stock solution and will be used both in Method A as well as in Method B. This solution shall be used within four days.

A-6.3.2 *Method A*

Take 100 ml of 0·1 percent dye solution in a 250-ml beaker and to this add two-three drops of phenolphthalein indicator. Titrate the solution with standard (0·1 *N*) sodium hydroxide solution from a burette till the solution turns slightly pink. Continue the titration and determine the end point when one or two drops of the solution taken from glass rod give for the first time a temporary blue colour reaction with external thymol blue indicator. (A porcelain spot plate is helpful for this titration.)

A-6.3.3 *Method B*

Take 100 ml of 0·1 percent dye solution in a 250-ml beaker and note the *p*H with the help of a *p*H meter. Titrate the solution with standard (0·1 *N*) sodium hydroxide solution. The end point is obtained by plotting the *p*H values on Y-axis and dv on X-axis. The end point is taken as the volume corresponding to second peak.

A-6.3.4 *Calculation*

$$\text{Acid value} = \frac{V \times N \times 56{\cdot}1}{M}$$

where

V = volume in ml required to neutralize the dye solution,

N = normality of standard alkali, and

M = mass in g of dye.

A-7 DETERMINATION OF DYE CONTENT

A-7.0 Outline of the Method

The scale of colorimeter is so designed that the scale reading is directly proportional to the concentration of the dye solution obeying Bouguer/Lambert-Beer Law.

A-7.1 Apparatus

Spectrophotometer with properly calibrated scale.

A-7.1.1 *Filters*

Green filters (K-S. 54, spectral range 500-570 nm).

A-7.1.2 *Solution Cells*

Sides of the cell shall be fused not cemented and cell must be resistant to acids, alkalis and other solvents and should have the following dimensions:

Thickness of glass	2 mm
Depth of cell	10 mm
Length of cell	40 mm
Height of cell	80 mm

A-7.2 Preparation of 0·002 Percent Dye Solution

First prepare 0·1 percent solution as follows:

Weigh accurately 0·1 g of lac dye in a 50-ml beaker. Add 10 ml of 95 percent ethyl alcohol and heat on a water bath for 10 minutes. Allow for sedimentation for 3-4 minutes and then decant the clear solution into a 100 ml measuring flask. Repeat this by taking further 6-8 ml of ethyl alcohol till dye goes into the solution. Allow the solution in the measuring flask to cool to room temperature and make the volume up to mark. Filter it under vacuum if necessary, through a sintered Gooch crucible/funnel into a thoroughly washed and dried filtering flask. Transfer the clear dye solution into a washed and dried reagent bottle.

Now take 2 ml of dye solution into a 100 ml measuring flask and make it up to mark with 95 percent ethyl alcohol.

This is stock solution (0·002 percent) and will be used for colorimetric as well as UV-visible spectrophotometric studies.

A-7.3 Procedure

Switch on the colorimeter and after the warming up period of the instrument set the instrument to zero for the cell filled with 9 percent ethyl alcohol. Remove the cell and replace the ethyl alcohol by the dye solution under examination. Before this wash the cell by test solution twice. Record the reading on the scale after usual working with the instrument.

A-7.4 Calculation

$$\text{Dye content, percent by mass} = \frac{X \times 0{\cdot}17 \times 10^{-4}}{0{\cdot}002}$$

where

X = reading in colorimeter.

A-8 DETERMINATION OF UV-VISIBLE ABSORPTION SPECTRUM

A-8.0 Outline of the Method

Laccaic acid contains different chromophoric groups which gives characteristic absorptions at different wavelengths in the UV-visible region. These absorptions are to be utilized for the standardization of lac dye.

A-8.1 Apparatus

a) Spectrophotometer (Grating type, range 200-700 nm);

b) Volumetric flask, pipettes, etc.

A-8.2 Reagent

Alcohol 95 percent conforming to IS 323 : 1959 provided that it is colourless.

A-8.3 Procedure

A-8.3.1 *Automatic Instrument*

Set the wavelength at 700 nm. Take a pair of cuvettes which are already matched at this wavelength. In one of the cuvettes, take the solvent ethyl alcohol. Wash the other cuvette twice with lac dye solution in ethyl alcohol of concentrated 0·002 percent, already prepared. Finally take the above lac dye solution in the cuvette. Scan for the absorbance at different wavelengths in the range 700-200 nm at a suitable chart speed. Compare the spectrum obtained with the standard spectrum of lac dye in ethyl alcohol. Check for the presence of absorption maximum at wavelengths 230 nm, 292 nm, 340 nm, 500 nm, and 530 nm.

A-8.3.2 *Manual Instrument*

Note the absorbance value at 700 nm and then the absorbance values at different wavelengths up to 200 nm, at wavelength intervals of 5 nm. Draw the spectrum (absorbance *vs* wavelength) on a graph paper. Check for the presence of absorption peak at the wavelengths 230 nm, 292 nm, 340 nm, 500 and 530 nm.

A-8.4 The sample shall be considered passing if its spectrum compares closely with that of the standard.

ANNEX B

(*Clause* 6.1)

SAMPLING OF LAC DYE

B-1 LOT

In a consignment all the containers of lac dye manufactured by a single source and same batch shall constitute a lot. Samples shall be taken and tested from each lot separately for ascertaining the conformity to the requirements of the specification.

B-2 Sample containers shall be taken at random from each lot as given in Table 2. For random selection procedures, reference may be made to IS 4905 : 1968.

B-3 The sample containers as given in col 2 of Table 2 shall be opened and sufficient material shall be collected from each of them. A part of the material collected from each container shall be thoroughly mixed to form a composite sample. All the characteristics given in Table 1 except dye content shall be tested on composite sample and in the case of dye content, the samples collected from individual containers shall be tested.

Table 2 Scale of Sampling

(*Clause* B-2)

No. of Containers in the Lot (1)	No. of Containers in the Sample (2)	No. of Tests for Dye Content (3)
Up to 150	3	1
151 to 300	4	2
301 and above	5	3

B-4 No failure shall occur if the lot is to be accepted under this clause.

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